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THREE-DIMENSIONAL MODEL FOR CALCULATING DURATION OF VISCOUS-FLOW SINTERING

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A method for estimating the duration of isothermal sintering of a powdered body and its final porosity and shrinkage is considered. The method, based on a three-dimensional model in the form of a cubic packing of spherical particles, uses sintering physics equations, the physicochemical properties of materials, and particle sizes. The model allows for describing not only the initial state (coalescence of particles) and the final state (compression of individual pores), but also the intermediate consolidation stage according to the general mass transfer mechanism. The sintering parameters for a sample of container glass powder are calculated using the viscous flow mechanism. The absolute discrepancy between the estimated results and experimental data is equal to 2.0% in porosity and 1.5% in shrinkage.

The quantitative description of the consolidation of powdered bodies in sintering is not only of theoretical interest but of great practical significance as well. The theoretical estimate of the duration of sintering powder preforms to a preset density makes it possible to determine the firing temperature and the interval of the sintered state for particular materials, as well as the effect of the phase composition and the structure of the powder. Such estimate can be obtained using a calculation method based on the physical regularities of mass transfer, whose parameters are the physicochemical properties of materials and the structural parameters of particles and their packing.

The main principles of the physics of sintering proposed in the works of Ya. I. Frenkel', G. S. Kuchinskii, B. Ya. Pines, Ya. E. Geguzin, et al. are summarized in [1]. The kinetics of sintering of various materials is described by uniform equations whose parameters are determined by the prevailing mechanism of mass transfer. For most mechanisms (diffusion across the volume of crystals, along the boundaries, along the surface, viscous flow, transfer via the gaseous phase, sintering under an external mechanical load) kinetic equations have been derived and experimentally validated and the conditions of their applicability have been identified. However, the use of these equations in practical calculations is limited, since they are based on unidimensional flat models and describe only the initial (coalescence of particles) and the final (compression of isolated pores) sintering stages. The intermediate stage of a decreasing volume of communi-

cating permeable pores in a contiguous solid-phase matrix has not yet been described. The known estimates [2] of isothermal sintering duration based on one-dimensional models differ from real values a thousand times.

In the context of the phenomenological approach, sintering can be described as the process of consolidation of a real powder body within the entire porosity variation interval [3]. The extension of this method has led to the development of methods for the mathematical modeling of sintering [4]. The parameters in the phenomenological equations are empirical ones determined experimentally. Lately attempts have been made to correlate the physical and the phenomenological approaches by estimating the parameters of the phenomenological equation based on the physicochemical properties of the sintering material [5].

In applied research the theoretical conclusions are mainly used qualitatively.

For estimating the total duration of the consolidation of a powder body using the equations of the physics of sintering, it is necessary to develop a unified physical model of the material structure at all stages of the process, including the intermediate stages, taking into account the volumetric packing of particles.

We have earlier [6] proposed a 3D model of a powder body, the sequence of the structure modification stages in sintering, and methods for calculating the duration of these stages based on kinetic equations of the physics of sintering. These methods were used to describe the diffusion sintering of corundum ceramics.

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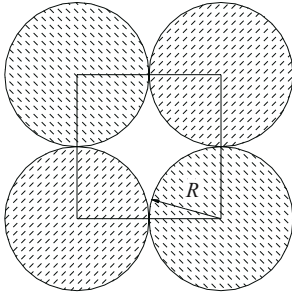


Fig. 1. Cubic packing of particles.

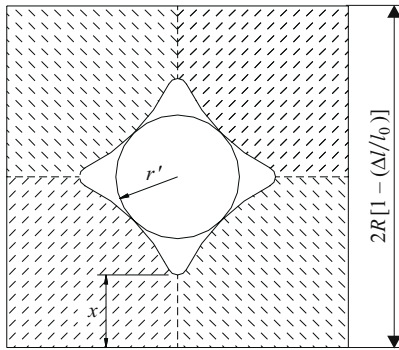


Fig. 2. Face of the cell of a sample at the stage of sintering [the vertices of the square correspond to the centers of particles; dashed lines) interparticle boundaries].

The purpose of the present study is to evaluate the suitability of this method for describing viscous-flow sintering.

Three-dimensional packing model and sequence of sintering stages. The following assumptions have been made:

- all particles are spherical of radius R ;
- the particles form an elementary cell that can be described as a regular polyhedron; the size of the cell is significantly smaller than the size of the body;
- the coordination number (CN) of particles in sintering does not change;
- long-range arrangement of particles is absent; i.e., the symmetric axes of the cell are oriented with respect to the symmetry axes of the body at different angles with equal probability; shrinkage is isotropic;
- the body structure is homogeneous;
- sintering in the entire porosity interval occurs by the same mechanism in isothermal conditions;
- sintering proceeds by the most favorable way; i.e., such undesirable phenomena as localization of sintering, coalescence of pores, etc. are absent.

The simplest 3D model of the packing of spherical particles is the cubic model (CN = 6) whose porosity (47.64%) agrees with the porosity of actual preforms made of narrow-fractionated powders (45 – 55%).

Figure 1 shows the section of a cubic cell across its face in the initial state. Parting from the geometry of the model

cell, the shrinkage along its edge (convergence of particle centers) is equal to

$$\Delta l/l_0 = 1 - \cos(\arcsin(x/R)), \quad (1)$$

where x is the radius of the contact bridge between the particles (Fig. 2).

The kinetics of the bridge growth is described by the exponential equation of the following form:

$$x^n - x_0^n = (\tau - \tau_0)f(A, \sigma, R^m, T), \quad (2)$$

where A is the mass transfer kinetic parameter; σ is the specific surface energy; τ is the time; T is the thermodynamic temperature; n and m are constants.

The beginning of sintering ($\tau_0 = 0$) corresponds to point contact ($x_0 = 0$).

Equation (2) in the case of a viscous flow has the following form [1]:

$$x^2 - x_0^2 = \frac{3}{2} \frac{\sigma R (\tau - \tau_0)}{\eta}, \quad (3)$$

where η is the viscosity of the material at the specified temperature.

The estimated dilatometric (macroscopic) shrinkage of the sample $\Delta L/L_0$ and the convergence of the particles centers in the cell $\Delta l/l_0$ are proportional:

$$\frac{\Delta L}{L_0} = K \frac{\Delta l}{l_0}. \quad (4)$$

In view of the assumption on the absence of the long-range order in the body structure, the coefficient K in the case of the simplest cubic packing is equal to the mean of the lengths of the cube edge and the cube diagonal, i.e., $\frac{1}{2}(1 + \sqrt{3}) \approx 1.366$.

Within a single cell volume, the volume of the solid phase is equal to $\frac{4}{3}\pi R^3$. The porosity of the sample is related to shrinkage:

$$Por = \frac{V_{\text{cell}} - V_{\text{sol}}}{V_{\text{cell}}} = \frac{\left[2R\left(1 - \frac{\Delta l}{l_0}\right)\right]^3 - \frac{4}{3}\pi R^3}{\left[2R\left(1 - \frac{\Delta l}{l_0}\right)\right]^3} = 1 - \frac{\pi}{6\left(1 - \frac{\Delta l}{l_0}\right)^3}. \quad (5)$$

At the first state of sintering, particles coalesce, i.e., their centers converge (shrinkage) and the surface area of the bridges between the particles grows.

According to expression (4), dilatometric shrinkage of 5.00% in the cubic packing corresponds to shrinkage within

a two-particle system of 3.66%, $x_1/R = 0.26808$ according to Eq. (1) and the porosity according to expression (5) is equal to $Por_1 = 0.414$ or 41.4%. After a transformation according to Eq. (3) it is possible to determine the duration of the first state τ_1 . A shrinkage equal to 5% is accepted, since according to the calculation, the total sintering duration in the linear shrinkage interval $\Delta L/L_0$ from 2 to 15% varies within the limits of $\pm 7\%$ of the process duration in relation to the shrinkage value at $(\Delta L/L_0)_1 = 5\%$.

By the moment τ_1 the maximum surface curvature is registered in the bridges between the particles and the sites around the interpore bridges ("the pore neck"). The curvature radius of the "pore neck" r' on the face of the cell with the edge length $2R[1 - (\Delta L/L_0)_1]$ connecting the central hollow spaces inside the neighboring cells (Fig. 2) is equal to

$$r' = \frac{R[1 - (\Delta L/L_0)_1]}{\cos(\pi/4)} - R. \quad (6)$$

At the second stage, the convergence of particle centers and the isolation of pores proceed independently. One needs to take into account the isolation of pores at this stage and their subsequent spheroidizing for transition from a system of contacting spheres of the solid phase to an ensemble of isolated pores in the solid matrix. The solid phase surface on the cell face has the maximum positive curvature, since the growing bridges of the neighboring particles are maximally close. The isolation of pores follows the mechanism of surface mass transfer.

Since other mass transfer mechanisms, apart from viscous flow, are absent from glass powder sintering, this process is also described by Eq. (3).

At the initial moment, a sphere of the radius r' can be inscribed into the pore neck (Fig. 2). At the end of the process the entire face of the cell exists in the solid phase of closely packed particles. The interface surface in the adjacent sites is formed by conjugated convex and concave surfaces of the particles and the emerging bridge. In the center of the face, slightly lower and slightly higher than the plane shown in Fig. 3, the solid phase has a spherical surface, concave toward the face plane.

Let us imagine that at the moment when the sealing of the pore neck ends, two spheres of the radius r' (Fig. 3) contact at a point in the center of the face. The spheres are hollow. The process of sealing of the pore neck can be represented as the coalescence of two hollow spheres in reverse time, i.e., at the initial conditions $x'_0 = r'$ with $\tau_0 = \tau'$ and the final conditions $x' = 0$ with $\tau = 0$. We replace the radius R in Eq. (3) by r' . The solution of this equation yields the value τ' .

By the end of sealing of the central pore neck $\tau_2 = \tau'$, the radius of the bridge according to Eq. (3) is equal to x_2 , the estimated dilatometric shrinkage according to formula (4) is $(\Delta L/L_0)_2$, and the porosity is Por_2 .

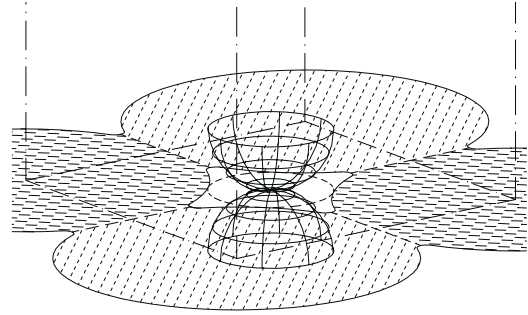


Fig. 3. Halves of hypothetical hollow spheres in the center of the bottom face of the cubic cell [dot-and-dash lines] cell edges].

We assume all pores to be sealed after the sealing of the pore necks (at the moment $\tau = \tau_1 + \tau_2$). Actually at the specified level of shrinkage and porosity some pores remain open.

Let us estimate the part of the interpore bridges. Each pore contacts six neighboring ones. The ratio of the volumes of the adjacent segments V_{segm} to the total volume of the pores is

$$\frac{6V_{\text{segm}}}{Por_2 [2R(1 - (\Delta L/L_0)_2)]^3} = 0.194.$$

According to the calculation $Por_2 \approx 30\%$ and the volume part of the interpore bridges is $0.3 \times 0.194 = 0.0582$, i.e., is relatively low; therefore, we ignore the error in describing the structure of these sites.

The structure of a sintering glass powder preform is characterized by the absence of boundaries between particles, i.e., we assume that after the isolation of pores the individuality of particles is lost and the pores are dispersed in the continuous glass matrix. Therefore, when estimating the shrinkage and consolidation of the body at the stages of spheroidizing and pore compression, one should take into account the increased system viscosity due to the inclusion of the second phase, namely, the pores. For this purpose we used the dependence of viscosity on the volumetric share of the dispersed phase C_v [7]:

$$\eta = \eta_0 \left(1 + \frac{K_c C_v}{1 - (C_v / C_{v \text{ cr}})} \right)^2, \quad (7)$$

where η_0 is the viscosity of the dispersion medium (material) at the specified temperatures; K_c is a constant depending on the shape and material of the dispersed phase inclusions; and $C_{v \text{ cr}}$ is the maximum content of the disperse phase in the system being deformed.

Let us assume $K_c = 2.5$ for isometric inclusions, $C_v = Por_2$, and $C_{v \text{ cr}} = \sqrt{2} \frac{\pi}{6} \approx 0.7405$, which corresponds to the degree of filling of the densest face-centered cubic packing of spheres.

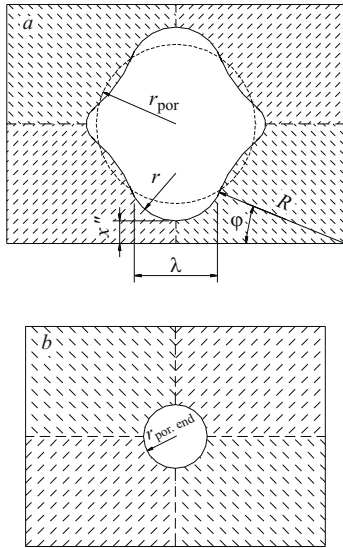


Fig. 4. Section of a cell by plane (101) at the stage of spheroidizing of the pore (a) and at the state of compression of the isolated pore (b).

The presence of the second phase inclusions does not affect the rate of changing of the interphase surface geometry but modifies the kinetics of shrinkage and consolidation at the later stages of sintering. Therefore, the duration of the pore insulation stage ($\tau_2 = \tau'$) and the spheroidizing stage τ_3 are calculated using the value η_0 in Eq. (3), which corresponds to the viscosity of pore-free glass:

$$x^2 - x_0^2 = \frac{3}{2} \frac{\sigma R (\tau - \tau_0)}{\eta_0}. \quad (8)$$

At the third stage of sintering, isolated pores become spheroidized due to the different curvature of the convex surfaces of particles directed inside the central pore and the concave surfaces in the area of the cell face center near the pore neck.

At the third stage, porosity and shrinkage are calculated based on the same equations as at the preceding consolidation stages.

The geometrical considerations (Fig. 4a) yield the following relations between the thickness of the interparticle spaces x'' , the distance between diagonally positioned particles λ , and the angle between the segment connecting the centers of the particle and the conjugacy point of the convex and concave surfaces of the interface φ :

$$x'' = R \sin \varphi - [R(1 - \sin \varphi) + \lambda/2] \frac{1 - \sin \varphi}{\cos \varphi}; \quad (9)$$

$$r = \frac{R(1 - \sin \varphi) + \lambda/2}{\cos \varphi}. \quad (10)$$

The maximum curvature is observed on the surface of the interparticle space (cup) between the particles located on the

lateral diagonal of the cell. Spheroidizing ends when its curvature radius r becomes equal to the pore radius r_{por} :

$$r_{\text{por}} = \left[\frac{V_{\text{por}}}{\frac{4}{3}\pi} \right]^{\frac{1}{3}} = \left[\frac{V_{\text{cell}} - V_{\text{sol}}}{\frac{4}{3}\pi} \right]^{\frac{1}{3}} = \left[\frac{8R^3 \left[1 - \left(\frac{\Delta l}{l_0} \right)_2 \right]^3 - \frac{4}{3}\pi R^3}{\frac{4}{3}\pi} \right]^{\frac{1}{3}}. \quad (11)$$

The radius r is determined based on Eqs. (9) and (10) using formula (8) after the calculation of x'' .

The solution of Eq. (10) with respect to φ at $r = r_{\text{por}}$ and $\lambda/2 = r'$ yields the value of the angle φ_{end} (the moment of the end of spheroidizing). The substitution of φ_{end} into expression (6) makes it possible to find the value x''_{end} and the solution of Eq. (8) for $x''_{\text{beg}} = 0$ and x''_{end} gives the duration of the pore spheroidizing stage τ_3 .

According to Eq. (3) with a correction for viscosity variation based on formula (7), within this time the value x grows to x_3 , which, considering expressions (4) and (5) corresponds to the shrinkage $(\Delta L/L_0)_3$ and the porosity Por_3 . Then the radius of spherical pores $r_{\text{por, sf}}$ is recalculated substituting $(\Delta l/l_0)_3$ in formula (11).

The structure of the sample at the beginning of the final stage of sintering represents a cubic packing of isolated spherical pores of the radius $r_{\text{por, sf}}$ in a continuous matrix. At this stage the pores are compressed under the effect of capillary pressure P_{cap} and external pressure, which are opposed by the gas pressure P_{gas} in the pore. The compaction ends at the moment of reaching their equilibrium. The final shrinkage, porosity, and pore size for an insoluble gas depends on its quantity contained in isolated pores.

The equilibrium state of spherical pores is characterized by the radius $r_{\text{por, end}}$ calculated by the Boyle – Mariott law, assuming the gas in the pore to be ideal (Fig. 4b).

For a cell with a single isolated pore the following equality holds:

$$V_{\text{sf}}^{\text{gas}} = \frac{P_3 V_3^{\text{gas}}}{P_{\text{eq}}} = \frac{4}{3} \pi r_{\text{por, end}}^3; \quad (12)$$

$$V_3^{\text{gas}} = V_3 \text{Por}_3,$$

where $V_3 = 8R^3 [1 - (\Delta l/l_0)_3]^3$ and Por_3 are the volume of the cell and the porosity at the moment of the end of pore spheroidizing; P_3 is the gas pressure at the moment of isolation equal to the pressure in the furnace.

The effect of external pressure on the value $r_{\text{por. end}}$ is neglected. In the state of equilibrium

$$P_{\text{eq}} = P_{\text{cap}} = \frac{2\sigma}{r_{\text{por. end}}}.$$

The equilibrium pore radius is equal to

$$r_{\text{por. end}} = \left(\frac{3}{8\pi} \frac{PV_3 \text{Por}_3}{\sigma} \right)^{\frac{1}{2}}. \quad (13)$$

The duration τ_4 of the final stage of compression of isolated pores from the radius r_{por} to $r_{\text{por. end}}$ is calculated based on the equation below, which is common for the cases of small and large P (compared with capillary pressure P_{cap}) [1]:

$$\tau_4 = \frac{2\eta_4}{P} \ln \left(\frac{2\sigma + r_{\text{por. sf}} P}{2\sigma + r_{\text{por. end}} P} \right), \quad (14)$$

where P is the external pressure; η_4 is the viscosity of the porous medium based on Eq. (7) at $C_v = \text{Por}_3$.

The total shrinkage of the model cubic cell is calculated based on formula (11) by substituting the value $r_{\text{por. end}}$, and the final porosity is found based on Eq. (5).

The total duration of sintering in isothermal conditions is equal to the sum of the durations of individual stages ($\tau_1 + \tau_2 + \tau_3 + \tau_4$).

Calculation of sintering duration. The material of the samples is clear bottle glass of the following composition (wt.%): 70.56 SiO₂, 0.32 Al₂O₃, ≤0.06 Fe₂O₃, 9.39 CaO, 3.32 MgO, 15.66 Na₂O, and 0.64 SO₃.

We assume the samples to consist of spherical particles of the radius $R = 10 \mu\text{m}$, the glass is a Newtonian fluid [8], its sintering proceeds in isothermal conditions in air under a pressure of 10^5 Pa , and the air is insoluble in the material.

The temperature dependence of glass viscosity [9] in the range of $10^{1.20} - 10^{4.97} \text{ Pa} \cdot \text{sec}$ at the temperatures of $800 - 1400^\circ\text{C}$ is approximated by the following polynomial:

$$\eta_0 = -4.9999 \times 10^{-9} t^3 + 2.28691 \times 10^{-5} t^2 - 3.7990 \times 10^{-1} t + 23.291.$$

The specific surface energy (surface tension) of glass at a temperature of 1300°C calculated according to the additivity rule [9] is $\sigma_{1300} = 0.318 \text{ J/m}^2$, its temperature dependence $\sigma(t)$ was assumed to be linear:

$$\sigma(t) = 0.318 - 3.18 \times 10^{-5} (t - 1300).$$

At the temperature of 700°C this glass is characterized by $\eta_0 = 1.5 \times 10^7 \text{ Pa} \cdot \text{sec}$, $\sigma = 0.337 \text{ N/m}^2$.

The duration of the first stage of coalescence at the temperature of 700°C corresponding to the linear shrinkage of samples $(\Delta L/L_0)_1 = 5\%$ and $\text{Por}_1 = 41.4\%$ according to

TABLE 1

Temperature, °C	Estimated parameters of glass powder samples with particle diameter, μm			
	5	10	20	30
<i>Duration of isothermal sintering, min</i>				
400	2.9×10^5	4.3×10^5	5.2×10^5	5.5×10^5
450	2.6×10^4	3.7×10^4	4.5×10^4	4.8×10^4
500	2.7×10^3	3.9×10^3	4.7×10^3	4.9×10^3
550	3.3×10^2	4.8×10^2	5.8×10^2	6.1×10^2
600	49.0	71.0	86.0	90.0
650	8.5	12.0	15.0	16.0
700	1.7	2.5	3.0	3.1
750	0.40	0.57	0.69	0.72
<i>Shrinkage $\Delta L/L_0$, %</i>				
400	25.92	24.86	21.98	18.46
750	25.86	24.78	21.77	18.08
<i>Porosity, %</i>				
400	1.6	4.3	11.4	19.1
750	1.7	4.5	11.9	19.8

Eqs. (1), (3), and (4) is equal to $\tau_1 = 0.36 \text{ min}$. The pore neck radius $r' = 3.63 \times 10^{-6} \text{ m}$.

The duration of the second stage (isolation of pores) is equal to $\tau_2 = \tau' = 0.66 \text{ min}$, the linear shrinkage of the sample is 14.7% , and the porosity $\text{Por}_2 = 26.4\%$.

The duration of the stage of spheroidizing $\tau_3 = 1.53 \text{ min}$. Taking into account formula (7), according to Eqs. (1), (3), (4), and (5) the end of the third stage corresponds to $(\Delta L/L_0)_3 = 20.5\%$; $\text{Por}_3 = 14.8\%$. After the recalculation, the radius of the spherical pores $r_{\text{por. sf}} = 7.10 \times 10^{-6} \text{ m}$.

At the moment of the end of sintering the equilibrium radius calculated based on formula (13) $r_{\text{por. end}}$ is equal to $5.11 \times 10^{-6} \text{ m}$. Taking into account expression (7) from Eq. (14), we obtain the duration of the pore compression stage $\tau_4 = 0.42 \text{ min}$.

Thus, according to the proposed calculation method, the total duration of isothermal sintering of a glass powder sample via the viscous flow mechanism at a temperature of 700°C is equal to about 3.0 min , the final linear shrinkage of the sample is 21.8% , and the porosity 11.8% .

Let us estimate the effect of the dispersion of powder on the estimated values of the final shrinkage and porosity, as well as the sintering duration at different temperatures (Table 1).

The pore volume distributed between the particles in sintering is related to the size of this particle by relations (5), (11), and (13). The weight of the gas contained in a pore at the end of the third stage depends on the size of the cell, i.e., on the size of the initial particles. The amount of gas in isolated pores is constant; therefore, the smaller the initial particle size, the smaller the volume of captured gas, the equilibrium pore radius, and the porosity of sintered material.

The viscosity of sintering material determines the duration of sintering. The effect of temperature on final shrinkage

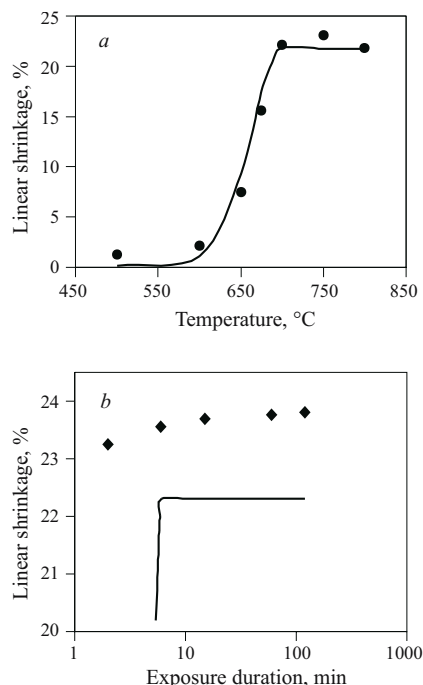


Fig. 5. Temperature dependence of the final shrinkage of samples (a) and shrinkage kinetics of glass samples at 700°C (b): dots) experimental data; solid curves) calculated values.

and porosity is determined by the weak temperature dependence of the specific surface energy of glass and is insignificant compared to the effect of the particle size. An increase in the pore size from 5 to 30 μm raises the duration of sintering by less than two times. The change in the shrinkage and porosity of sample within the firing temperature interval of 400 – 750°C does not exceed the experimental error in determining these parameters.

Experimental validation of the method. The experiment was performed on glass powder of the specified composition ground by wet milling in a ball mill until completely passing through a sieve with a cell size of 40 μm . The fraction of 8 – 12 μm with a mean particle radius of 10.4 μm was separated by sedimentation. Disk-shaped samples of diameter 16 mm and height 2 – 3 mm were molded at a pressure of 20 MPa.

The porosity of the preform was 44.3%, which is close to the density of the simplest cubic packing.

The method of consecutive firings in an electric furnace in air (Fig. 5a) was used to investigate the temperature dependence of shrinkage. The heating rate was 2.5 K/min. The exposure duration at the maximum temperature was 6 min including the estimated duration of heating the sample in the

mold, which at 500 – 800°C was equal to 2.8 – 3.2 min. Cooling was performed together with the furnace.

The kinetics of sintering was studied at the temperature of 700°C (Fig. 5b). The process mainly ends within a time period close to the theoretical estimate, taking into account the preform heating period ($\tau \approx 6$ min). When the exposure was extended to 120 min, the shrinkage of the samples grew by no more than 0.6%. The difference between the estimated and experimental shrinkage is not more than 1.5%.

The difference between the experimental isothermal sintering duration and the duration calculated using the above method is no more than tens of percent, whereas calculations based on the one-dimensional model yield a duration value that is a thousand times longer.

The experimental values of porosity (10.3%) and shrinkage (23.1%) of samples after firing with 6 min exposure at 700°C agree satisfactorily with the calculated values of 12.3% and 21.6%, respectively. The deviations are related to the fact that the porosity of the actual preforms (44.3%) is somewhat lower than the porosity of the model cubic cell (47.6%).

Thus, the proposed model and the calculation method based on this model adequately describe sintering of glass powder samples by the viscous flow mechanism in isothermal conditions. The calculation makes it possible to estimate the duration of isothermal sintering, the final shrinkage, and the porosity with an accuracy sufficient for practical use.

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